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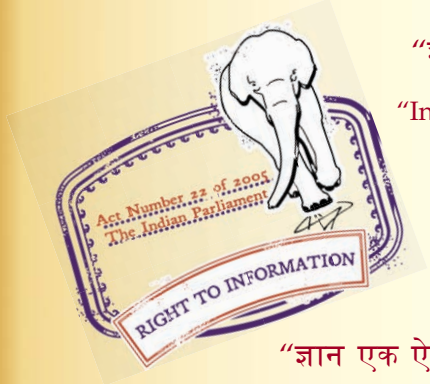
IS 6515 (1999): Sodium Pentachlorophenate [PCD 9: Organic Chemicals Alcohols and Allied Products and Dye Intermediates]



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IS 6515 : 1999

(Reaffirmed 2004)

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सोडियम पेन्टाक्लोरोफिनेट — विशिष्टि

(पहला पुनरीक्षण)

Indian Standard

SODIUM PENTACHLOROPHENATE — SPECIFICATION

(First Revision)

ICS 71.080.99

BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

March 1999

Price Group 4

Organic Chemicals Miscellaneous Sectional Committee, PCD 9

FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Organic Chemicals Miscellaneous Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

Sodium pentachlorophenate (C_6Cl_5ONa), is mainly used as a molluscicide. It may also be used as weedicide, fungicide, preservative, white ant-repellant and as a fermentation disinfectant. It has also been found useful in products like guar gum.

This standard was first published in 1972 as sodium pentachlorophenate, technical. The Committee responsible for its preparation decided to revise it as sodium pentachlorophenate and not as sodium pentachlorophenate, technical since over the years the usage of this material as weedicide or fungicide has reduced considerably in favour of more advanced products and it has found better usage as industrial preservative, specially for wood and leather treatment. In this first revision, the methods of test for determination of chlorophenates, moisture and the free alkalinity have been modified.

For the purpose of deciding whether a particular requirement of this standard is complied with the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

SODIUM PENTACHLOROPHENATE — SPECIFICATION

(First Revision)

1 SCOPE

This standard prescribes the requirements and the methods of sampling and test for sodium pentachlorophenate.

2 NORMATIVE REFERENCES

The following Indian Standards contain provisions which through reference in this text constitute the provisions of the standard. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

IS No.	*Title*
323 : 1959	Specification for rectified spirit
1070 : 1992	Water for general laboratory use (*third revision*)
1260 (Part 2) : 1979	Pictorial markings for handling of good: Part 2 General goods (*second revision*)
4161 : 1967	Specification for Nessler cylinders

3 REQUIREMENTS

3.1 Description

The material shall be buff coloured powder granules or in the form of solution.

3.2 The material shall comply with the requirements given in Table 1.

4 PRECAUTIONS IN STORING AND HANDLING

Sodium pentachlorophenate is a poison, interfering with phosphorylation and oxidation processes in metabolism. Penetration into the body is possible through respiratory and digestive organs. It is advisable not to smoke, eat or drink after handling sodium pentachlorophenate without first washing face and hands with great care. Persons should be provided with waterproof gloves of rubber or plastic material, spectacles and antidust mask while testing this dangerously poisonous material. It is harmful to expose wounds to sodium pentachlorophenate dusts.

Table 1 Requirements for Sodium Pentachlorophenate
(*Clauses* 3.2, 7.2, E-6.1 *and* E-6.2)

Sl No.	Characteristic	Requirement	Method of Test (Ref to Cl No. in Annex)
(1)	(2)	(3)	(4)
i)	Chlorophenate (as C_6Cl_5ONa), percent by mass, *Min*	80	A
ii)	Moisture, ml/100 g, *Max*	16	B
iii)	Free alkalinity (as NaOH), percent by mass	0.2 to 0.5	C
iv)	Colour of the aqueous solution	To pass the test	D

5 PACKING AND MARKING

5.1 Packing

The material shall be packed in clean, dry and air-tight containers made of mild steel sheets, tin plate, fibre board, wood or in double hessian bags with polyethylene lining.

5.2 Marking

The containers shall be securely closed and shall bear legibly and indelibly the following information:

a) Name of the material;
b) Name of the manufacturer and/or his recognized trade-mark, if any;
c) Lot or batch number;
d) Net mass of the contents; and
e) Minimum cautionary notice worded as under:

POISON: AVOID CONTACT WITH SKIN. DO NOT BREATHE DUST OR FUME. KEEP WELL AWAY FROM FOOD STUFFS, EMPTY FOOD STUFF CONTAINERS AND ANIMAL FEED.

NOTE — When hessian bags are used for packing this material, the pictorial marking for 'USE NO HOOKS' as specified in IS 1260 (Part 2) shall be stencilled on the bags.

5.2.1 *BIS Certification Marking*

The container may also be marked with the Standard Mark.

5.2.2 The use of Standard Mark is governed by the provisions of the *Bureau of Indian Standards Act*,

1986 and the Rules and Regulations made thereunder. The details of conditions under which the licence for the use of the Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

6 SAMPLING

Representative samples of the material shall be drawn and their conformity to this standard shall be judged as prescribed in Annex E.

7 METHOD OF TEST

7.1 Quality of Reagents

7.1.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

7.2 The material shall be tested for all requirements as given in Table 1 and according to the methods specified in Annexes A to D.

ANNEX A

[*Table* 1, *Sl No.* (i), and *Clause* 7.2]

DETERMINATION OF CHLOROPHENATES

A-1 PRINCIPLE

Sodium pentachlorophenate is dissolved in water and chlorophenols precipitated by acidification with sulphuric acid. The chlorophenols are filtered, extracted with acetone and titrated to end point by standard sodium hydroxide solution with phenolphthalein as an indicator.

A-2 REAGENTS

A-2.1 Dilute Sulphuric Acid, 25 percent (*v/v*).

A-2.2 Acetone AR

A-2.3 Phenolphthalein Indicator Solution

A-2.4 Standard Sodium Hydroxide Solution, 0.1 N.

A-3 PROCEDURE

Take accurately about 1 g of sample or 10 ml sample by volumetric pipette in beaker. Add about 100 ml distilled water. Shake well to dissolve it. Then add 25 ml dilute sulphuric acid. Heat the solution on preheated water bath at 50 to 60°C for digestion for half an hour. Cool to room temperature and filter it on asbestos padding with the help of vacuum pump. Wash the precipitate with water until the filtrate is neutral to litmus paper. Avoid excess washings. Transfer the precipitate and asbestos pulp in same beaker with help of glass rod. Add 200 ml acetone. Dissolve the precipitate in acetone. Titrate with standard sodium hydroxide solution using phenolphthalein as an indicator. End point will be colourless to pink. Carry out a blank test.

A-4 CALCULATION

$$\text{Chlorophenates (as } C_6Cl_5ONa\text{), percent by mass} = \frac{(V - B) \times N \times 28.85}{M}$$

where

V = volume in ml of standard sodium hydroxide solution required for titration of the material;

B = volume in ml of standard sodium hydroxide solution required for blank titration;

N = normality of standard sodium hydroxide solution; and

M = mass in g of material taken for the test.

ANNEX B

[*Table* 1, *Sl No.* (ii), and *Clause* 7.2]

DETERMINATION OF MOISTURE

B-1 GENERAL

For the determination of moisture content the distillation method has been specified.

B-2 APPARATUS

B-2.1 Distillation Flask

Made of hard glass with 500 ml capacity and provided with a ground-glass socket.

B-2.2 Reflux Condenser

Made of glass, water cooled having the outside diameter of the inner tube 16 to 17 mm, and that of the jacket 23 to 25 mm. The lower end of the condenser is provided with a ground glass cone. The tip of the condenser is ground at an angle of approximately 30° from the vertical axis.

B-2.3 Receiver (or Trap)

Made of hard glass and consisting essentially of an upper chamber, provided with a side-arm leading to the distillation flask, and a cylindrical graduated portion, the lower end of which is sealed. The opening of the upper chamber is provided with a ground-glass socket fitting the cone of the condenser. The lower end of the side-arm provided with a ground glass cone fitting the socket of the distillation flask. The graduated portion has a capacity of 5 ml when filled to the highest graduation mark. The scale covers the range 0 to 5 ml, with graduation marks at 0.1 ml intervals. The error at any indicated capacity should not exceed 0.02 ml.

B-2.4 Heat Source

Either an oil-bath or an electric heater provided with a sliding rheostat or other means of heat control. The temperature of the oil in the bath should not be very much higher than the boiling point of the petroleum naphtha (*see* **3.2.2**).

B-2.5 Copper Wire

Long enough to extend through the condenser with one end twisted into a spiral. The diameter of the spiral should be such that it fits snugly within the graduated portion of receiver and yet can be moved up and down.

B-3 REAGENTS

B-3.1 Chromic Acid Cleaning Solution

B-3.2 Solvents

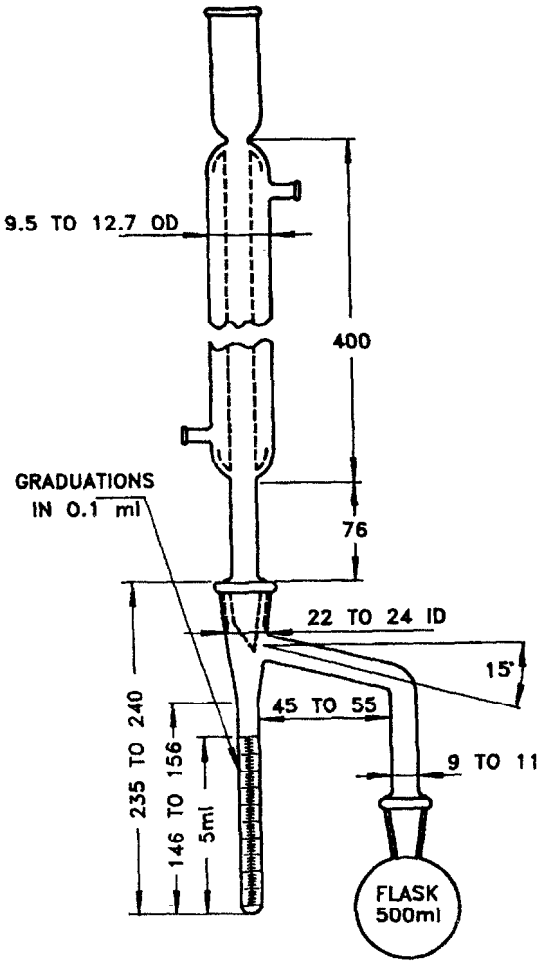
One of the following solvents may be used:

a) *Petroleum Naphtha* — with a boiling range of 90 to 210°C;
b) n-*Heptane* — boiling point around 98°C; and
c) *Toluene* — boiling point around 110°C.

B-4 PROCEDURE

Clean the entire apparatus (*see* Fig. 1) with chromic acid solution to minimize the adherence of water droplets to the sides of the condenser and the receiver. Rinse thoroughly with distilled water and dry completely before using.

Place an appropriate amount of the sample accurately weighed into the distillation flask. The amount of sample taken should be such that the distillation flask is not more than one third full, it should not contain more than 1.8 ml of water. Add sufficient volume of the solvent (100 to 200 ml) so that the sample shall be completely covered by the solvent while the distillation is in progress. Swirl to mix and add pumic stone to ensure steady boiling. Assemble the apparatus and fill the receiver with the solvent by pouring the solvent through the condenser until it begins to overflow into the distillation flask.



All dimensions in millimetres.

FIG. 1 MOISTURE DISTILLATION APPARATUS

Attach a guard tube containing anhydrous calcium chloride at the top of the condenser to prevent condensation of atmospheric moisture within the tube. In order that the reflux may be under control, wrap the distillation flask and the tube leading to the receiver with asbestos fibre. Heat the flask at such a rate that about 100 drops are distilled over, increase the distillation rate to about 200 drops/min and continue heating until no more water is collected. Purge the condenser occasionally during the distillation with 5 ml portions of the solvent in order to wash down any moisture adhering to the walls of the condenser. The water in the trap may be made to separate from the solvent by means of a copper wire, one end of which is twisted into a helix. The copper wire if moved up and down in the condenser and receiver occasionally causes the water to settle down at the bottom of the trap. Reflux until the water level in the receiver remains unchanged for 30 minutes and then shut off the source of heat. Flush the condenser with the solvent, making use of the copper wire to disengage

any moisture droplets. Allow to stand for about 15 minutes and then read the volume of water.

B-5 CALCULATION

$$\text{Moisture (ml/100 g)} = \frac{V \times 100}{M}$$

where

V = volume in ml of water collected in receiver, and

M = mass in g of the material taken for the test.

ANNEX C

[*Table* 1, *Sl No.* (iii), and *Clause* 7.2]

DETERMINATION OF FREE ALKALINITY

C-1 OUTLINE OF THE METHOD

Acidimetric titration of aqueous solution of sodium pentachlorophenate using thymolphthalein indicator.

C-2 REAGENTS

C-2.1 Standard Sulphuric Acid, 0.1 N.

C-2.2 Thymolphthalein Solution

Dissolve 0.1 g of thymolphthalein in 150 ml rectified spirit (*see* IS 323) and add 100 ml water with stirring.

C-3 PROCEDURE

Weigh accurately about 2 g of the material and dissolve it in 100 ml of distilled water in a 500-ml conical flask. Add 5 drops of thymolphthalein solution. Titrate with standard sulphuric acid until the colour changes from the blue to colourless, while carefully crushing with a glass rod the precipitate formed during neutralization.

C-4 CALCULATION

$$\text{Free alkalinity (as NaOH), percent by mass} = \frac{4.0\,VN}{M}$$

where

V = volume in ml of standard sulphuric acid required for the titration,

N = normality of standard sulphuric acid, and

M = mass in g of the material taken for the test.

ANNEX D

[*Table* 1, *Sl No.* (iv), and *Clause* 7.2]

DETERMINATION OF COLOUR OF THE AQUEOUS SOLUTION

D-1 OUTLINE OF THE METHOD

A weighed quantity of the material is dissolved in water and the colour developed, if any, is compared with that of the 8 percent (*m/v*) solution of anhydrous ferric chloride.

D-2 APPARATUS

D-2.1 Nessler Cylinder, two, 100 ml (*see* IS 4161).

D-3 REAGENTS

D-3.1 Ferric Chloride Solution

Dissolve 16.0 g of anhydrous ferric chloride in 200 ml of water.

D-4 PROCEDURE

Dissolve 8 g of the material in water. Transfer the solution to a Nessler cylinder and make up the volume with water to 100 ml mark. Take 100 ml of the ferric chloride solution in the other Nessler cylinder and compare the colour of the two solutions.

The material shall be regarded as having passed the test if the intensity of colour of the aqueous solution is not deeper than that of the ferric chloride solution.

ANNEX E

(*Clause* 6)

SAMPLING OF SODIUM PENTACHLOROPHENATE

E-1 GENERAL

E-1.1 Samples shall be taken at a place protected from damp air, dust and soot.

E-1.2 Sampling instrument shall be clean and dry.

E-1.3 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

E-1.4 To draw a representative sample, the contents of each container, selected for sampling, shall be mixed, as thoroughly as possible, by suitable means.

E-1.5 The samples shall be placed in clean, dry and air-tight glass or other suitable containers on which the material has no action.

E-1.6 The sample containers shall be of such a size that they are almost completely filled by the sample.

E-1.7 Each sample container shall be sealed air-tight after filling and marked with full details of sampling, the date of sampling, year of manufacture and other important particulars of the consignment.

E-1.8 Samples shall be stored in a cool and dry place.

E-2 SAMPLING INSTRUMENT

E-2.1 The sampling instrument is a closed-type sampling tube, undivided (*see* Fig. 2) consisting of two concentric cylindrical tubes made of a metal which is not affected by the material being sampled (preferably of stainless steel) closely fitting into each other throughout their entire length so that it is possible to rotate one tube within the other, a suitable handle being provided for the purpose. Longitudinal openings of about one-third the circumference are cut in both tubes throughout their length. In one position the two openings coincide and admit the material into the hollow inner tube. By rotating the inner tube through 180°, the opening is tightly closed and a 'core' of material being enclosed therein may be withdrawn. This type of sampler is usually provided with a locking arrangement so that the tubes are held together in any desired position. The outer tube is provided with a sharp conical end to facilitate penetration but the base of the cone shall be closed so that no material is entrapped in this portion. The height of the cone shall be equal to its base diameter. The whole instrument shall be of sufficient length to penetrate an entire diagonal of the container being sampled. The diameter of the inner cylindrical space may vary from 20 to 40 mm proportionately to the length. A length of 150 cm and a diameter of 30 mm can cater for most of the needs.

E-2.1.1 *Use of Sampling Instrument*

The instrument is inserted in closed position in a oblique direction till it touches bottom. The material is admitted by rotating and opening the tubes and finally closing them, withdrawing the sample in this process. In case the minimum quantity of material required for test from each container is more than the capacity of the instrument, further 'cores' shall be taken from different parts of the same containers such that they are at least 75 mm in the case of drums, bags, etc, and 25 mm in the case of small containers, from the wall of the container. In all cases the instrument shall be inserted till it touches bottom so that an entire cross-section is withdrawn.

E-3 SCALE OF SAMPLING

E-3.1 Lot

All the containers in a single consignment of the material drawn from a single batch of manufacture shall constitute a lot. If a consignment is declared or known to consist of containers pertaining to different batches of manufacture, the containers belonging to the same batch of manufacture shall be grouped together and each such group shall constitute a separate lot.

E-3.2 For ascertaining the conformity of the lot to the requirements of this specification, tests shall be carried out for each lot separately. The number of containers (n) to be selected for drawing the samples shall depend upon the size of the lot (N) and shall be in accordance with Table 2.

Table 2 Number of Containers to be Selected for Sampling

Lot Size	No. of Containers to be Selected
(N)	(n)
(1)	(2)
4 to 25	3
26 " 50	4
51 " 100	5
101 " 150	6
151 " 300	7
301 and above	8

NOTE — When the size of the lot is three or less, all the containers shall be sampled.

E-3.3 These containers shall be selected at random from the lot and to ensure the randomness of selection, random number tables shall be used. In case such tables are not available, the following procedure may be adopted:

Starting from any container, count them in one order as 1, 2, 3....., up to *r* and so on, where *r* is the integral part of *N*/*n* (*see* **E-3.2**). Every *r*th container thus counted shall be withdrawn to give sample for test.

E-4 TEST SAMPLE AND REFEREE SAMPLE

E-4.1 From each of the container selected as in **E-3.2**, draw with an appropriate sampling instrument small portions of the material from different parts of the container. The total quantity so drawn from each of the containers shall be approximately equal to thrice the quantity required for testing purposes.

E-4.2 Mix thoroughly all the portions of the material drawn from the same container to give a representative sample for the container.

E-4.3 From the samples (*see* **E-4.2**) representing different containers selected in **E-3.2**, a small but equal quantity of material shall be taken and thoroughly mixed to form a composite sample, sufficient to carry out testing for the characteristics specified. The composite sample so obtained shall be divided into three equal parts, one for the purchaser, another for the suppler and the third for the referee.

E-4.4 The remaining portion of the material in the samples (*see* **E-4.2**) from different containers shall be divided into three equal parts, each forming an individual sample. One set of individual samples representing the *n* containers selected shall be for the purchaser, another for the supplier and the third for the referee.

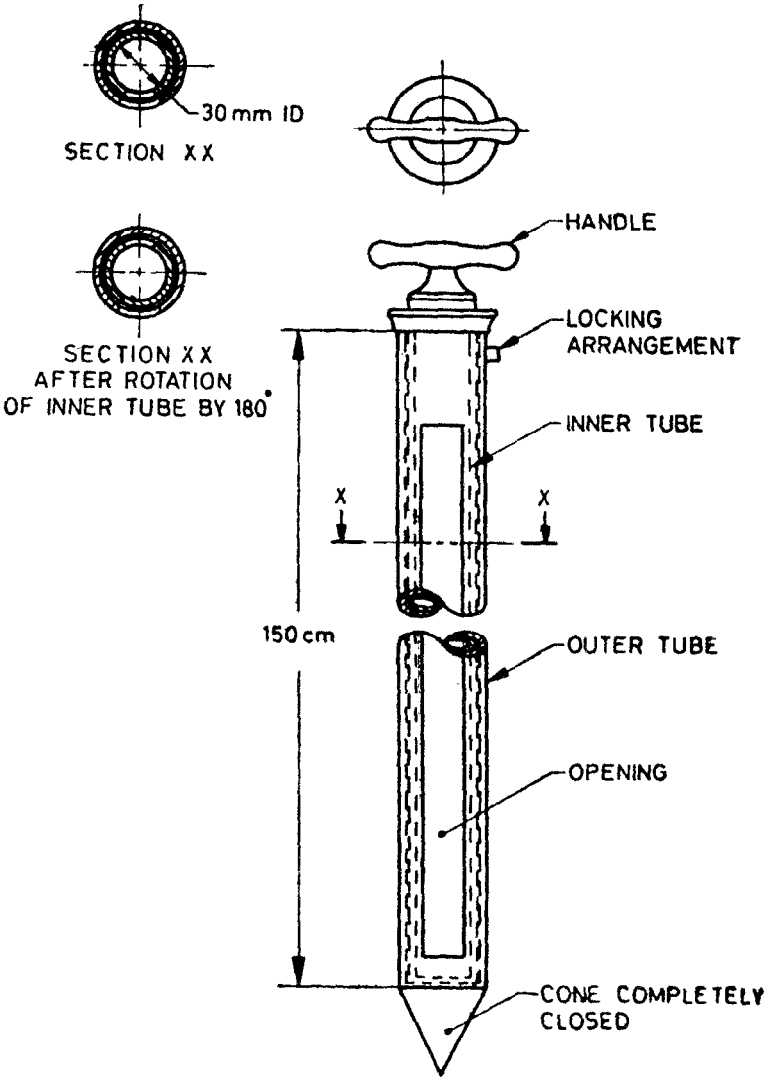


FIG. 2 CLOSED TYPE SAMPLING TUBE, UNDIVIDED

E-4.5 All the individual and composite samples shall be transferred to separate containers. These containers shall then be sealed air-tight with stoppers and labelled with full identification particulars given in **E-1.7**.

E-4.6 The referee samples consisting of a composite sample and a set of *n* individual samples, shall bear the seals of both the purchaser and the supplier and shall be kept at a place agreed to between the two. This shall be used in case of any dispute between the two.

E-5 TESTS

E-5.1 Tests for description (*see* **3.1**) and determination of chlorophenates shall be conducted on each of the individual samples.

E-5.2 Tests for the remaining characteristics shall be conducted on the composite sample.

E-6 CRITERIA FOR CONFORMITY

E-6.1 For Individual Samples

The lot shall be declared as conforming to the requirements of description and chlorophenates if each of the test results satisfy the corresponding requirements given under **3.1** and Table 1.

E-6.2 For Composite Sample

For declaring the conformity of a lot to the requirements of all other characteristics (*see* **E-5.2**) tested on the composite sample, the test results for the characteristics shall satisfy the relevant requirements given in Table 1.

Bureau of Indian Standards

BIS is a statutory institution established under the *Bureau of Indian Standards Act*, 1986 to promote harmonious development of the activities of standardization, marking and quality certification of goods and attending to connected matters in the country.

Review of Indian Standards

Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the latest issue of 'BIS Handbook' and 'Standards: Monthly Additions'.

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Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

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